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ORIGINAL

Shape recovery and anomalous swelling of steam-compressed wood by swimming ring-like expansion of cell lumina

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Abstract To investigate the mechanism of shape fixation of steam-compressed wood, Japanese cedar wood specimens were compressed radially by 50% using saturated water vapor at 160 °C and observed during shape recovery in different liquids, including water and dimethyl sulfoxide (DMSO). Shape recovery was evaluated based on the dimensional changes in wet conditions (SR_w) and those in dry conditions (SR_d). SR_w in DMSO was significantly greater than that in water, but this recovery observed in DMSO was not recognized in SR_d. The contradiction arose from significant reversible cellular deformation: the compressed cells were reversibly expanded with the swelling of the cell wall, and this swimming ring-like cellular deformation increased SR_w in DMSO. At room temperature, the SR_d values in various liquids were closely connected to the swelling of the wood cell walls, rather than to the degree of softening. This suggested that the plastic expansion of cells, as well as the softening of the wood cell walls, caused the shape recovery of compressed wood. This mechanism was qualitatively expressed by an elasto-plastic model equipped with an inflator representing the swimming ring-like expansion of the cell lumina.

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Introduction

Because wood is a honeycomb-like material, its cell walls can be tightly folded under compression in the radial (*R*) or tangential (*T*) directions. Transverse compression, or densification, is a useful method to convert lightweight and soft coniferous lumber into a denser, stiffer, and harder material (Seborg and Stamm 1941; Inoue et al. 1990, 1993; Ito et al. 1998a; Dwianto et al. 1998; Kutnar et al. 2009; Laine et al. 2013). The biggest problem in wood densification is shape recovery. Although plastically deformed wood remains unrecovered in dry conditions, almost complete recovery occurs when the compressed wood is moistened, wetted, or boiled in water. Therefore, many researchers since the 1940s have attempted to prevent the shape recovery of densified wood. Among various shape fixation methods, heat treatment is the simplest and probably the most cost-efficient. Seborg and Stamm (1941) and Seborg et al. (1956) found that the compressed shape of wood could be fixed by oven heating. Later investigations by Inoue et al. (1993) suggested greater efficacy in shape fixation could be achieved by compression under saturated water vapor. Because this steam-compression method provides both faster shape fixation and minimal color change, it is widely used for densification and the three-dimensional molding of wood (Ito et al. 1998a; Navi and Girardet 2000). Recent developments in thermo-hydro-mechanical treatment have been reviewed by Sandberg et al. (2013).

Despite the prominence of the method, the mechanism of shape fixation by steam compression remains contested. Inoue et al. (1993) speculated that stress relaxation and the hydrophobization of amorphous matrix polymers were major mechanisms underlying shape fixation. However, Ito et al. (1998b) reported that shape fixation was accompanied by a significant increase in the crystallinity of cellulose during steam compression. They also confirmed that shape recovery did not occur when large amounts of matrix polymers were depolymerized and lost. From those results, Ito et al. concluded that shape fixation was predominantly driven by morphological changes in cellulose, rather than by structural changes in the amorphous matrix polymers. This interpretation was widely accepted, but later questioned by Higashihara et al. (2000). They found that the shape of steam-compressed wood could be recovered upon exposure to various organic liquids, such as dimethylsulfoxide (DMSO), even when the compressed shape was retained in boiling water. They speculated that the formation of hydrophobic structures upon the loss of hydrophilic hemicelluloses caused shape fixation.

Steaming involves the loss of hemicelluloses and cross-linking reaction in lignin (Tjeerdsma et al. 1998; Wikberg and Maunu 2004; Guo et al. 2015, 2017). These changes reduce the hygroscopicity of wood, and the reduced hygroscopicity prevents hydrothermal softening of amorphous wood polymers. Therefore, the hydrophobization due to steaming must contribute somewhat to the shape fixation of compressed wood. If the hydrophobization is the major mechanism, the shape of wood should be fixed by compression "after" steaming. However, such pre-steaming does not allow perfect shape fixation unless the wood is additionally heated for a longer duration (Inoue et al. 2008). Thus, hydrophobization is not sufficient to explain shape fixation by steaming.

The mechanism of shape recovery is also unclear. According to Inoue et al. (1993), compressive strain became temporarily fixed for moistened wood dried under compression (drying set), because intermolecular hydrogen bonds were reformed in the amorphous matrix polymers and the glassy-state frozen matrix retained the shape of the wood from dry conditions. In this case, shape recovery in boiling water is attributed to the plasticization and thermal softening of amorphous matrix polymers. However, the compressive strain of toluene-swollen acetylated wood is also set by the removal of toluene under compression, and the compressed shape is recovered upon toluene addition, whereas toluene has no hydrogen-bonding ability and its softening effect is less than that of water (Obataya and Yamauchi 2005). This indicates that the drying set and shape recovery of compressed wood cannot always be explained by the scission and reformation of intermolecular hydrogen bonds and the softening of matrix polymers.

In this study, the focus was put on the shape recovery of steam-compressed cedar wood. First, it was attempted to reproduce the experiment of Higashihara et al. (2000) to verify the hypothesis of the formation of hydrophobic structures inducing shape fixation. Next, the anomalous swelling of the steam-compressed wood was observed, involving the expansion of the cell lumen, which may be another mechanism contributing to shape recovery. Finally, a mechanical model was proposed to explain the shape fixation and recovery of steam-compressed wood.

Materials and methods

Steam compression of wood

Japanese cedar wood (*Cryptomeria japonica* D. Don) was cut into blocks with dimensions of 29 (longitudinal, *L*)×29 (tangential, *T*)×29 mm^3 (radial, *R*). The specimen density in dry conditions was 343 kg/m^3 (340–349 kg/m^3). These specimens were soaked in water at 25 °C under reduced pressure. The water-swollen specimens, measuring 32 mm in the *R* direction, were then compressed in the *R* direction by 15 mm (53%) in an autoclave at 160 °C for 15, 30, 60, 90, 120, and 240 min. Figure 1 shows the autoclave used for the steam compression. The inner volume of the autoclave was 1400 cm^3 (20×20×3.5 cm^3). At 160 °C, the autoclave was filled with saturated water vapor and the internal pressure was maintained

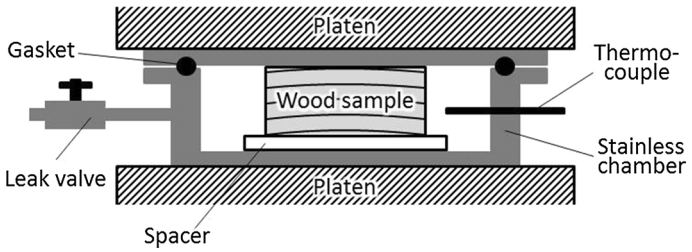

Fig. 1 Autoclave used for steam compression

throughout steam compression. After steam compression, the leak valve was opened to release the pressurized water vapor and the autoclave was immediately cooled to 100–105 °C. The wood specimens were then dried in the autoclave for more than 12 h; afterward, their dry masses and dimensions were measured. Nine specimens were used for each treatment condition.

Recovery testing

The steam-compressed specimens were soaked in water at 25 °C under reduced pressure. Some of the water-swollen specimens were subsequently boiled in water at 95 °C for 1 h for shape-recovery observations. After the recovery treatments, the specimens were air-dried at 25 °C and 60% relative humidity (RH) for more than 1 week, followed by oven drying at 105 °C for 1 day to permit measurement of the recovered dimensions in dry conditions.

Next, the recovered specimens were soaked in DMSO at 25 °C under reduced pressure for 1 week to measure their DMSO-swollen dimensions. The specimens were then soaked in ethanol for 1 week, with ethanol refreshed each day, to replace the DMSO in the specimens with ethanol. After solvent exchange, the specimens were leached in running water for more than 3 days, air-dried, and finally oven-dried at 105 °C to determine their dry dimensions.

Three steam-compressed specimens from each treatment condition were divided into smaller specimens measuring 5 (L)×29 (T)×12–29 mm (R). These specimens were oven-dried to determine their dimensions in dry conditions; subsequently, they were subjected to recovery testing using one of the following 11 liquids: toluene (TL); 1,4-dioxane (DX); 1-propanol (PR); ethyl acetate (EA); acetone (AC); ethanol (ET); methanol (MT); water (CW); ethylene glycol (EG); pyridine (PD), and DMSO. The wood specimens were soaked in these liquids at 25 °C for more than 1 month and then dried completely to determine their recovered dimensions.

Swelling tests of recovered wood

After shape recovery in boiling water and DMSO, the wood specimens were dried to determine their recovered dimensions. Next, the recovered specimens were boiled in water and DMSO again to determine their normal (i.e., reversible) volumetric swelling. In addition, the cross sections of the specimens were observed using an optical microscope in dry and wet conditions.

Compression testing

Uncompressed cedar wood was cut into specimens with dimensions of 15 (L)×15 (T)×15 mm (R). These cubic specimens were conditioned at 25 °C and 60% RH, soaked in 25 °C water, boiled in 95 °C hot water, or soaked in 25 °C DMSO. The soaked or boiled specimens were then compressed in the R direction at a compression rate of 1 mm/min using a universal testing machine equipped with a water bath

to determine their compressive Young's moduli in the R direction (E_R). The E_R values were extracted from the linear parts of the obtained stress–strain curves.

Results and discussion

Shape recovery of steam-compressed wood

Figure 2 shows the effects of steaming duration on the loss in mass by steam compression and subsequent boiling in water. Prolonged steaming induces greater mass loss because of the thermal degradation of the wood constituents, predominantly the accelerated hydrolysis of hemicelluloses in the presence of moisture (Higashihara 2007; Sandberg et al. 2013).

Figure 3 illustrates the dimensional changes of the wood under steam compression and subsequent recovery treatments. Shape recovery of the compressed wood is generally evaluated by the strain recovery in dry or wet conditions, SR_d or SR_w, as defined by the following equations, respectively:

$$SR_d \equiv \frac{t_{d2} - t_{d1}}{t_{d0} - t_{d1}} \quad (1)$$

$$SR_w \equiv \frac{t_{w2} - t_{w1}}{t_{w0} - t_{w1}} \quad (2)$$

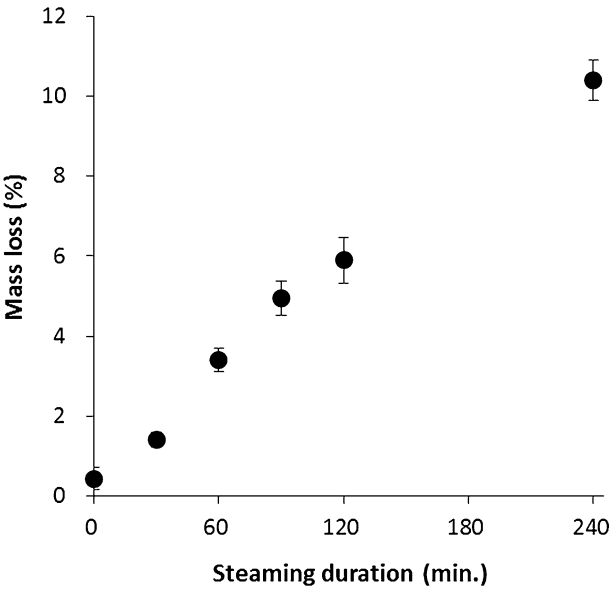

Fig. 2 Effects of steaming duration on the mass loss of wood during steam compression and subsequent boiling in water. Bars indicate standard deviations

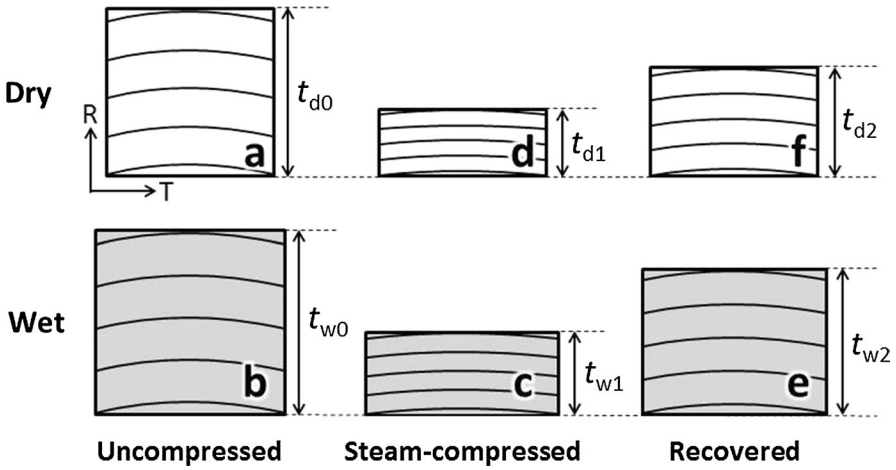

Fig. 3 Dimensional changes of wood during steam compression and subsequent recovery treatment

Both the SR_d and SR_w values should be 1 for perfect shape recovery (no fixation), and 0 for perfect shape fixation (no recovery). Figure 4 shows the SR_d values of the steam-compressed wood as a function of steaming duration. The SR_d value is decreased with increased steaming durations, and the compressed shapes are almost

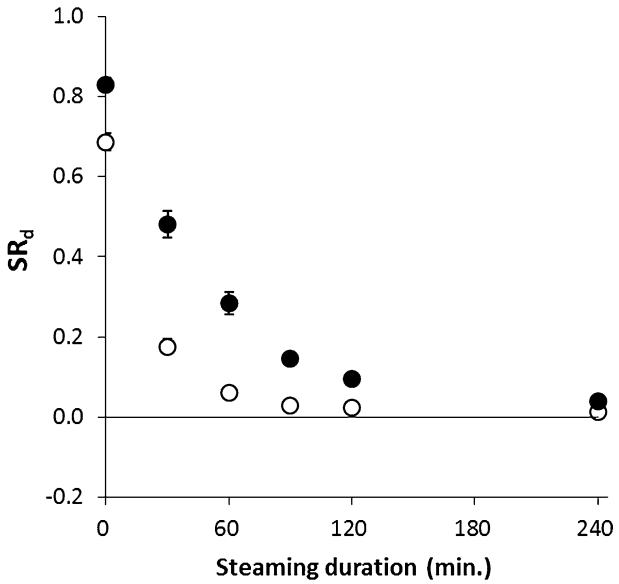

Fig. 4 Strain recovery of steam-compressed wood determined in dry conditions (SR_d) as a function of steaming duration. Open plots, strain recovery upon boiling in water; filled plots, strain recovery upon soaking in DMSO. Bars indicate standard deviations

completely fixed within 90 min: the SR_d value in boiling water approaches zero. Regarding the SR_d value, the additional recovery in DMSO was slight.

Figure 5 shows the SR_w values of steam-compressed wood as a function of steaming duration. Compared to the SR_d values, the SR_w values after soaking in DMSO are considerably greater. Similar considerable recovery was reported by Higashihara et al. (2000). They speculated that the formation of hydrophobic structures caused the shape fixation of compressed wood and that the significant increase in SR_w reflects the dissipation of this hydrophobic structure in DMSO, an aprotic organic liquid. As exhibited in Fig. 4, however, additional recovery by soaking in DMSO does not significantly affect the SR_d values. This contradictory result requires us to consider the unusual swelling behaviors of steam-compressed wood.

Inflation of cell lumen by wetting

In Fig. 6, the specific swelling of steam-compressed wood in the *R* direction is plotted against the steaming duration. The specific swelling $\Delta R/\gamma$ is defined as the radial swelling of wood (ΔR) divided by its specific gravity (γ) in dry conditions. If the cell lumen volume remains unchanged, $\Delta R/\gamma$ should be constant irrespective of the wood density. As shown in Fig. 6, the $\Delta R/\gamma$ value is remarkably increased with increasing steaming duration reaching 60–90 min; for further increases in duration, the value is decreased. The maximum value of $\Delta R/\gamma$ is 2–3 times greater than that of unmodified wood. The unusual swelling of the steam-compressed wood is not

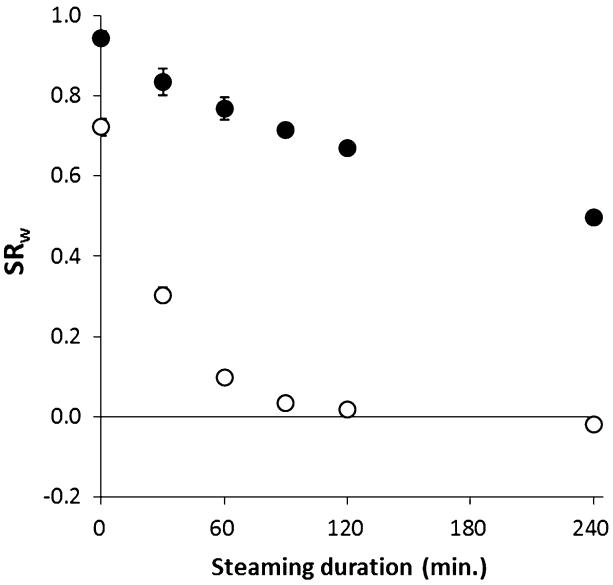

Fig. 5 Strain recovery of steam-compressed wood determined in wet conditions (SR_w) as a function of steaming duration. Open plots, strain recovery upon boiling in water; filled plots, strain recovery upon soaking in DMSO. Bars indicate standard deviations

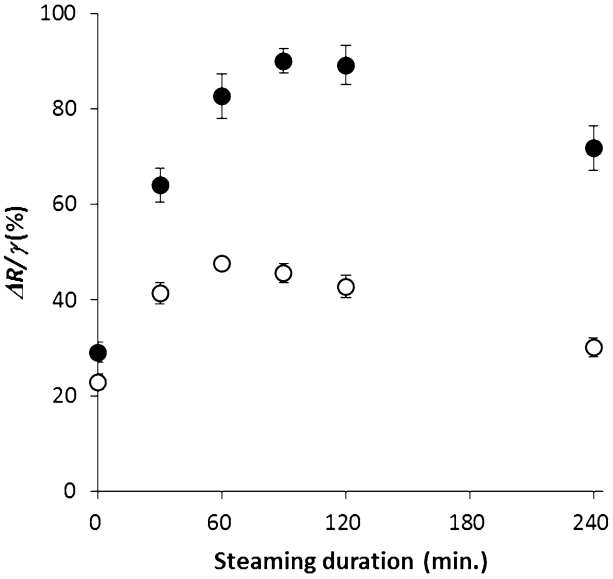

Fig. 6 Specific swelling ($\Delta R/\gamma$) of steam-compressed wood in R direction plotted against the steaming duration. Open plots, specific swelling in water; filled plots, specific swelling in DMSO. Bars indicate standard deviations

an effect of irreversible shape recovery, because the tested specimens have already experienced recovery by boiling in water and soaking in DMSO. In addition, the greater swelling of steam-compressed wood contradicts its reduced accessibility: the equilibrium moisture content of wood at 25 °C and 60% RH (desorption process) decreases monotonically from 12.6 (untreated) to 11.0% (steamed for 240 min) with increases in steaming duration. That is, the steam-compressed wood should show smaller $\Delta R/\gamma$ values if its reduced accessibility is taken into consideration. Thus, the marked swelling of the steam-compressed wood must be attributed to the swelling of the cell lumen. Figure 7 shows the cross sections of earlywood cells in a specimen that has been steam-compressed for 90 min. Steam compression has caused the earlywood cells to buckle tightly and fold in dry conditions, but these cells are expanded in the R direction by wetting and shrunk by drying. Similar "reversible" cellular deformation was reported by Miyoshi et al. (2016), but the mechanism has not yet been determined.

The reversible cellular deformation exhibited in Fig. 7 recalls the inflation and collapse of a swimming ring: a folded ring is inflated by pumping air into the tube, allowing recovery of the fully swollen shape, while removal of air from the tube causes shape collapse. In uncompressed wood, the cell lumen remains almost unchanged throughout wetting and drying because the cell shape is maintained by the rigid cell wall. However, after the cell wall has buckled and undergone tight folding by transverse compression, it is weakened by micro-failure at the folded corner, which allows the swimming ring-like deformation via swelling (pumping air into the tube) and shrinkage (removal of air from the tube). This mechanism seems

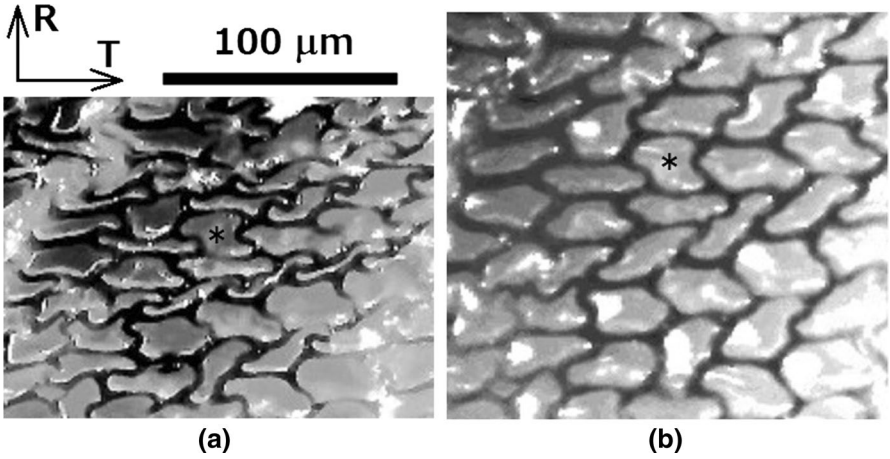

Fig. 7 Cross sections of steam-compressed earlywood cells in **a** oven-dried and **b** water-swollen conditions. Asterisks indicate the same cell

to explain the remarkable and reversible swelling of steam-compressed wood exhibited in Fig. 7.

The reversible cellular deformation of steam-compressed wood can also explain the considerable increase in SR_w upon soaking in DMSO (Fig. 5). Since the SR_w includes the reversible swelling of the cell lumen as well as irreversible shape recovery, SR_w shows an apparent increase for the temporary expansion of the cell lumen in DMSO. This shows that SR_w is not a good indicator of irreversible shape recovery for wood treated with certain liquids that cause remarkable cell wall swelling, such as DMSO.

The swimming ring-like deformation also explains the slight increase in SR_d upon soaking in DMSO (Fig. 4). By steaming for 90 min or longer, the compressed shape does not recover in boiling water; however, in DMSO, the wood cells show remarkable plastic inflation. Consequently, the expanded cell lumen remains unrecovered after the removal of DMSO, corresponding to a slight increase in SR_d.

Mechanisms of shape recovery

Figure 8 visualizes the mechanism of shape recovery as described by Inoue et al. (1993). For moistened wood dried under compression, the shape is temporarily set by the stress relaxation and rigidification of the amorphous matrix substances, while the hydrophobic and elastic microfibrils retain a certain elastic energy. For moistened wood with a softened amorphous matrix, the compressed shape is recovered by the release of the elastic energy stored in the microfibrils. This model explains the shape recovery of wood compressed or bent in hot water, but not the marked shape recovery observed in DMSO.

If the release of elastic energy stored in the microfibrils is a major driving force for shape recovery, greater recovery should be achieved with greater softening of the amorphous matrix. Figure 9 shows the SR_d values of unheated-compressed

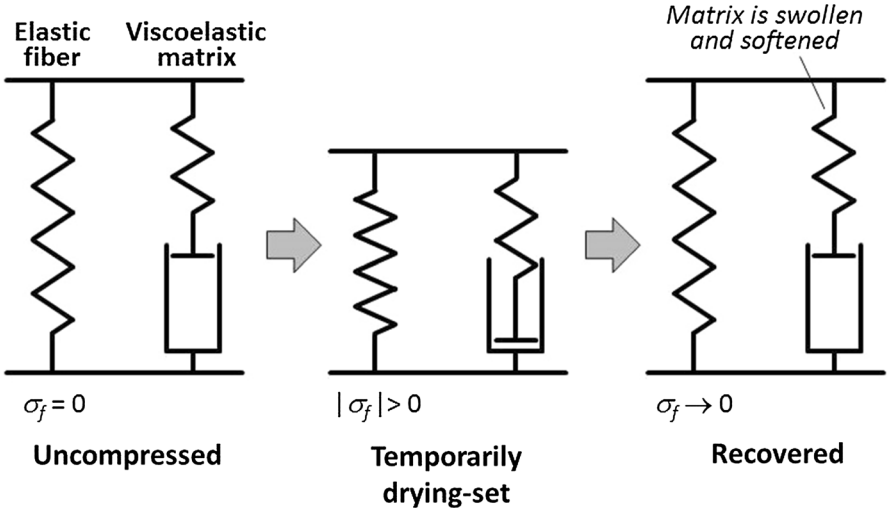

Fig. 8 A conventional viscoelastic model explaining the drying-set and recovery of wood

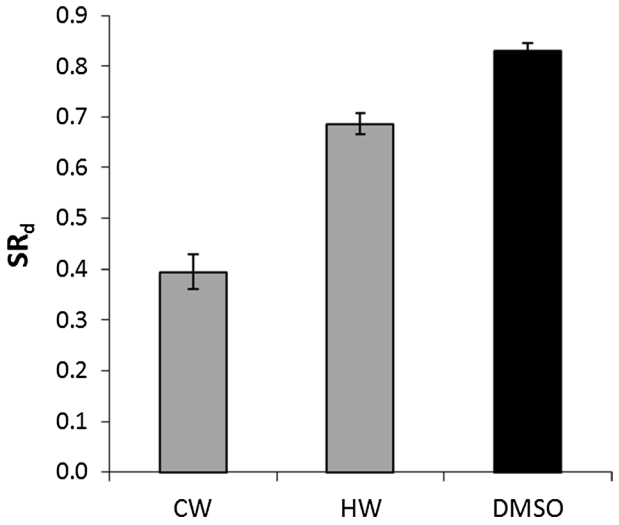

Fig. 9 Strain recovery (SR_d) of compressed wood in cold water (CW) at 25 °C, hot water (HW) at 95 °C, and DMSO at 25 °C

wood in cold water (CW), hot water (HW), and DMSO, and the Young's moduli (E_R) of uncompressed wood in these liquids are exhibited in Fig. 10. As predicted from the conventional model shown in Fig. 8, the shape recovery in HW is greater than that in CW because of the thermal softening of amorphous matrix. However, the conventional model does not account for the largest shape recovery occurring in DMSO, in which the softening of wood is less than that in HW.

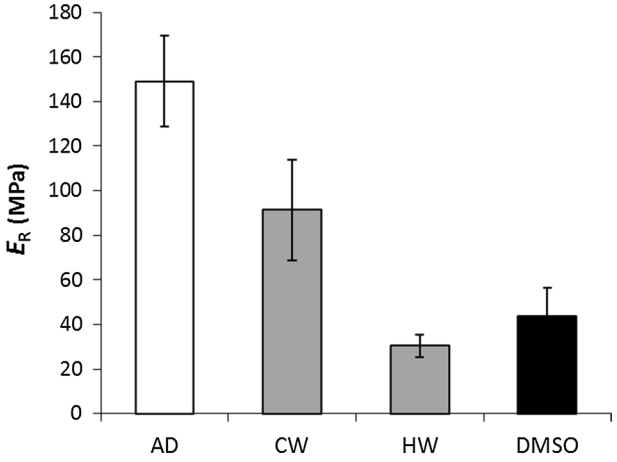

Fig. 10 Compressive Young's moduli (E_R) of uncompressed wood in *R* direction. Bars indicate standard deviations

It is considered that the folded cells in compressed wood are anomalously inflated upon cell wall swelling, as a folded swimming ring is. In this case, the swelling pressure in the wood cell wall is one possible driving force. If such inflation contributes to shape recovery, the degree of recovery should depend on the swelling of the cell wall. Figure 11 shows the volumetric swelling of steam-decompressed

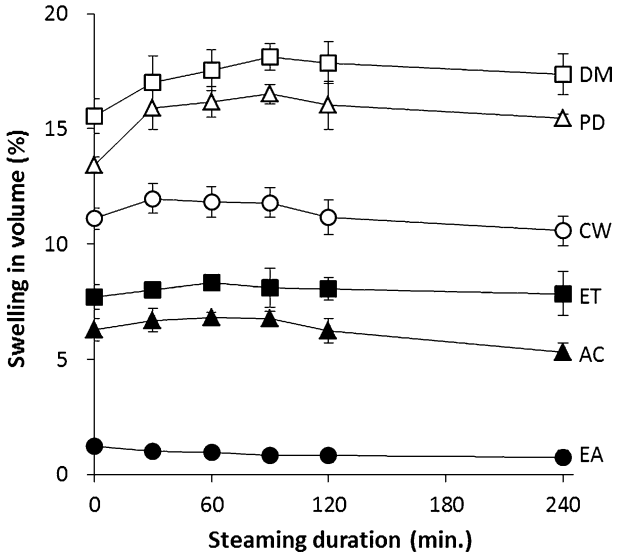

Fig. 11 Volumetric swelling of uncompressed wood in ethyl acetate (EA), acetone (AC), ethanol (ET), water (CW), pyridine (PD), and dimethyl sulfoxide (DM) at 25 °C plotted against the steaming duration. Bars indicate standard deviations. Data for toluene-, 1,4-dioxane-, 1-propanol-, ethanol-, methanol-, and ethylene glycol-treated uncompressed wood is omitted for visual clarity

wood specimens in various liquids. For visual clarity, data for specimens treated by liquids that induced very little swelling are omitted from the figure. The swelling is slightly increased as the steaming duration is increased to 90 min, before starting to decrease. The slightly greater swelling of the steamed wood is probably from the loosening of the fiber-matrix structure in the cell walls, as suggested by Rautkari and Hill (2014), but this effect is negligibly smaller than the broad variation in the degree of wood swelling in different liquids. In Fig. 12, the SR_d values of unheated and steam-compressed wood in various liquids are plotted against the volumetric swelling of uncompressed wood. The SR_d values simply depend on the volumetric swelling of wood, irrespective of the chemical natures and softening abilities of the liquids. This implies that the inflation of the cell lumen with cell wall swelling significantly affects the shape recovery of wood. The steam-compressed wood shows no shape recovery in aprotic organic liquids, whose swelling abilities are less than that of water, but it is recovered to some extent in pyridine and DMSO, which induce greater swelling of wood than water does. This fact also implies that the swelling of wood is an important factor affecting the shape recovery.

Figure 13 shows a hypothetical elasto-plastic model to explain both the irreversible shape recovery and reversible inflation of cells. Because no time-dependent phenomena are included, here a plastic slider is used instead of a viscous dashpot (Fig. 13a). Upon compression, the amorphous matrix is deformed plastically while some elastic energy remains in the fibers. In addition, a potential inflator appears upon the buckling of the cell wall (Fig. 13b). When the compressed wood is moistened or wetted, its shape is recovered by (1) the softening of the amorphous matrix, involving the release of elastic energy stored in the fiber, and (2) the activation of the inflator, i.e., the swimming ring-like inflation of cells, with the swelling of the cell wall (Fig. 13c). These changes are all irreversible because no driving force remains after the shape recovery. By steaming (Fig. 13d), the stress in the elastic fiber is

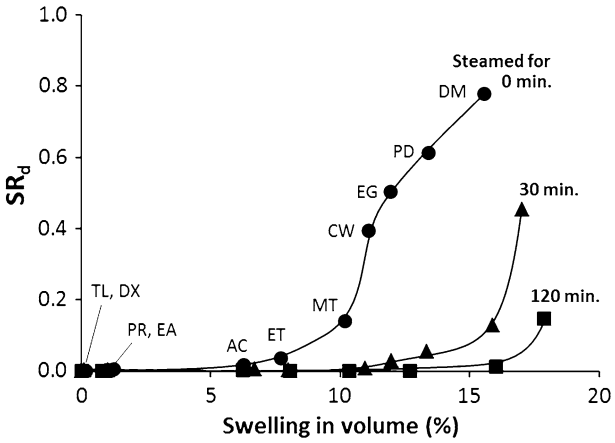

Fig. 12 Strain recovery (SR_d) of steam-compressed wood in different liquids at 25 °C plotted against the volumetric swelling of steamed uncompressed wood in the liquids. *TL* toluene, *DX* 1,4-dioxane, *PR* 1-propanol, *EA* ethyl acetate, *AC* acetone, *ET* ethanol, *MT* methanol, *CW* water, *EG* ethylene glycol, *PD* pyridine, *DM* dimethyl sulfoxide. Bars indicate standard deviations

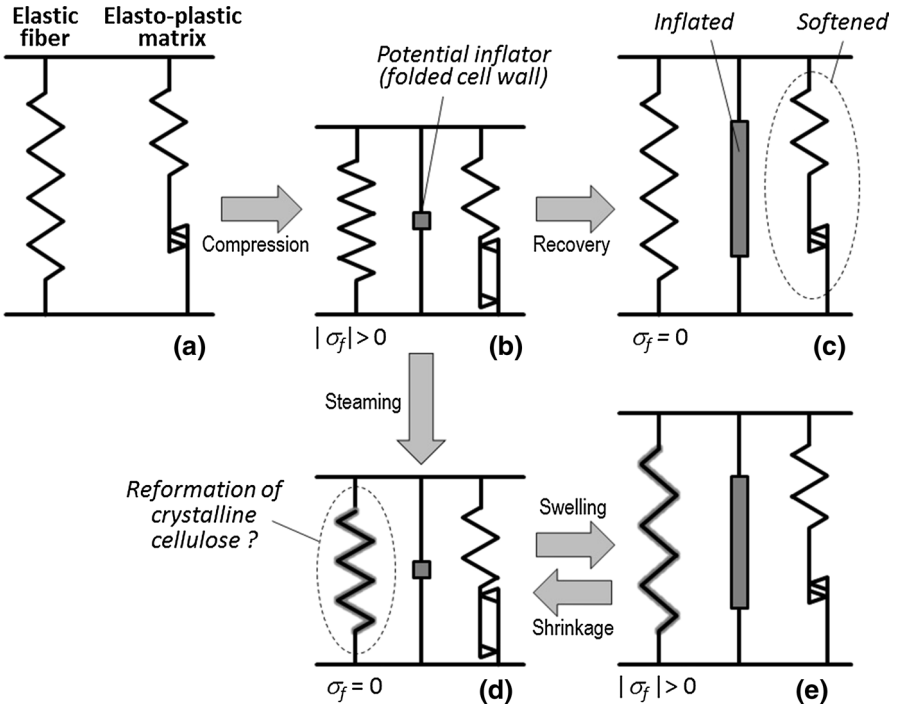

Fig. 13 An elasto-plastic model to explain **a**–**c** the irreversible shape recovery of compressed wood and **d**–**e** the reversible swelling and shrinkage of steam-compressed wood involving cellular deformation

relaxed, probably by the reformation of crystalline cellulose and/or the formation of cross-linking among the amorphous matrix polymers. Consequently, the compressed shape remains unrecovered upon boiling in water and soaking in DMSO. Even in this case, however, the inflator can be activated by the cell wall swelling itself; therefore, the steam-compressed wood shows anomalous dimensional changes because of the reversible expansion and shrinkage of the cell lumen.

When the presence of an inflator that represents swimming ring-like cellular deformation is assumed, both the irreversible shape recovery and anomalous swelling of compressed wood are explained without any contradictions. It is speculated that the swelling pressure is the driving force of such inflation, but further experiments are needed to connect the swelling pressure in the wood cell wall and the mechanical behaviors of the folded cell wall.

Conclusion

When evaluating shape recovery based on dimensional changes in wet conditions, steam-compressed wood showed more significant shape recovery in DMSO. However, this apparent recovery mainly arose from the remarkable and reversible expansion of the cell lumen in DMSO. The degree of shape recovery could not be

explained by the degree of softening: the shape recovery in DMSO was greater than that in boiling water, despite the greater softening induced by boiling water. In addition, the degree of shape recovery in different liquids depended strongly on the swelling of the cell wall, irrespective of the chemical nature of the liquids. These results indicated that the swelling of the wood cell wall, as well as its softening, significantly affected the shape recovery of compressed wood. These phenomena could be explained by an elasto-plastic model that considered swimming ring-like inflation of the buckled cell walls.

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